

भारतीय मानक

विद्युत्तरोधी द्रव — पोलychlorinated बाईफिनायलों (पी.सी.बी.)
द्वारा संदूषण — केपिलरी कॉलम गैस क्रोमेटोग्राफी
द्वारा ज्ञात करने की विधि

Indian Standard

INSULATING LIQUIDS — CONTAMINATION BY
POLYCHLORINATED BIPHENYLS (PCBs) —
METHOD OF DETERMINATION BY CAPILLARY
COLUMN GAS CHROMATOGRAPHY

ICS 13.020; 17.220.99; 29.035.40

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NATIONAL FOREWORD

This Indian Standard which is identical with IEC 61619 : 1997 'Insulating liquids — Contamination by polychlorinated biphenyls (PCBs) — Method of determination by capillary column gas chromatography' issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of the Fluids for Electrotechnical Applications Sectional Committee and approval of the Electrotechnical Division Council.

The text of IEC Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminology and conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
- b) Comma (,) has been used as a decimal marker, while in Indian Standards the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appear to the following International Standard for which Indian Standard also exists. The corresponding Indian Standard, which is to be substituted in its place is listed below along with its degree of equivalence for the edition indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
IEC 60475 : 1974 Method of sampling liquid dielectrics	IS 6855 : 2003 Method of sampling for liquid dielectrics	Technically Equivalent

Only the English language text has been retained while adopting it in this Indian Standard and as such the page numbers given here are not the same as in the IEC Publication.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

INSULATING LIQUIDS — CONTAMINATION BY
POLYCHLORINATED BIPHENYLS (PCBs) —
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1 Scope and object

This International Standard specifies a method for the determination of polychlorinated biphenyl (PCB) concentration in non-halogenated insulating liquids by high-resolution capillary column gas chromatography using an electron capture detector (ECD).

The method gives the total PCB content and is especially useful when a detailed analysis of PCB congeners is necessary. Other methods, such as IEC 60997, may be used when a less detailed analysis is acceptable.

The method is applicable to unused, reclaimed (including dechlorinated and chemically and/or physically treated), or used insulating liquids contaminated by PCBs.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this normative document. At the time of publication, the edition indicated was valid. All normative documents are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative document indicated below. Members of IEC and ISO maintain registers of current valid International Standards.

IEC 60475: 1974, *Method of sampling liquid dielectrics*

3 Definitions

For the purposes of the International Standard, the following definitions apply:

3.1 Polychlorinated biphenyl (PCB)

A biphenyl substituted by one to ten chlorine atoms.

NOTE – For legal purposes, congeners with one, two or ten chlorine atoms may be excluded from this definition.

3.2 Congener

All the chlorine derivatives of biphenyl, irrespective of the number of chlorine atoms, are termed congeners.

NOTE – There are 209 possible PCB congeners. These are listed in table B.1. The congener numbers (IUPAC)* are for easy identification; they do not represent the order of chromatographic elution.

* International Union of Pure and Applied Chemistry.

4 Principle

The PCB congeners are determined by temperature programmed gas chromatography. The chromatograph uses a high-efficiency capillary column to separate the PCBs into single or small groups of overlapping congeners.

The sensitivity of the electron capture detector (ECD) may be reduced by the presence of mineral oil. In this method the sample is diluted 100 times to reduce this effect to a minimum.

A sample preparation (clean-up) procedure is used to remove most of the impurities likely to interfere with the determination.

Reference compounds are included to enable calculation of experimental relative retention times (ERRTs) which are compared to a data file of peak ERRTs to identify individual or groups of unresolved congeners. An internal standard is added for quantification.

Relative response factors (RRF), taken from the data files (9.1), corrected by experimental relative response factors (ERRFs) obtained from reference compounds, are applied to identified peaks to quantify the individual (or groups of) congeners, and the values summed to give total PCB content.

5 Reagents and auxiliary materials

5.1 Reagents and standards

All reagents and materials, including those for clean-up, shall be free from PCB contamination and compounds responding to the ECD.

5.1.1 Solvent

Hexane, heptane, cyclohexane or isooctane (2,2,4-trimethylpentane), high purity, free from PCB contamination and low in compounds that respond to the ECD.

5.1.2 Hexachlorobenzene

Purity 99 % at least, used for checking detector sensitivity.

5.1.3 Insulating liquid

An insulating liquid, checked to be free from PCBs or other interfering substances, of the same type as is present in the sample.

5.1.4 Congener 30 solution (C 30)

10 mg/l in solvent (5.1.1) purchased in solution or prepared from pure material (purity 99 % at least).

5.1.5 Congener 209 (DCB), decachlorobiphenyl solution

10 mg/l in solvent (5.1.1) purchased in solution or prepared from pure material (purity 99% at least).

5.1.6 Calibration solution of selected PCB congeners

Certified calibration mixture in solvent (5.1.1) containing at least the following PCB congeners at a concentration of 10 mg/l each: 18, 28, 31, 44, 52, 101, 118, 138, 149, 153, 170, 180, 194 and 209 (see B.3).

5.2 Commercial PCB standards (see B.4)

5.2.1 Solutions of Aroclors® 1242, 1254 and 1260 in solvent (5.1.1)

Concentration required 50 mg/l or more, typically 1000 mg/l.

5.2.2 Solutions of Aroclors® 1242, 1254 and 1260 in oil

50 mg/kg solutions of Aroclors® 1242, 1254 and 1260 in unused insulating liquid, either purchased as standardized solutions or prepared from pure material.

5.3 Gas chromatography gases

5.3.1 Carrier gas: helium or hydrogen, purity 99,99 % at least.

5.3.2 Make-up gas: argon/methane, 95 % / 5 %. Alternatively, 99,99 % minimum purity nitrogen can be used.

5.4 Internal standard/reference solutions

NOTE – Standards should be stored in a cool, dark place.

5.4.1 Internal standard solution 2 (IS 2)

2 mg/l C209 (DCB), 2 mg/l C30.

Pipette (5.8.3) 5 ml of DCB solution (5.1.5) and 5 ml C30 solution (5.1.4) into a 25 ml volumetric flask, make up to the mark with solvent (5.1.1).

5.4.2 Internal standard solution 0,5 (IS 0,5)

0,5 mg/l C209 (DCB), 0,5 mg/l C30.

Follow 5.4.1 using a 100 ml volumetric flask.

5.5 Test mixture solution (for system evaluation)

Into a 20 ml volumetric flask: weigh, to the nearest 0,001 g, 0,50 g of 50 mg/kg Aroclor® 1260, plus 0,50 g of 50 mg/kg Aroclor® 1254 plus 1,00 g of 50 mg/kg Aroclor® 1242 solutions in insulating liquid (5.2.2).

Add by pipette 1 ml of IS 2 solution (5.4.1) and make up to volume with solvent.

Prior to use this solution shall be treated as per 11.1.3.

5.6 Calibration – congener mix stock solution

Into a 20 ml volumetric flask: weigh, to the nearest 0,001 g, 2,0 g of insulating liquid (5.1.3) and add 1 ml of the calibration PCB congener mix (5.1.6). Make up to the mark with solvent (5.1.1).

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5.7 Congener mix calibration solution (for response factors)

Submit 500 µl of solution (5.6) to the clean-up (11.1.3). The final solution is suitable for the determination of relative factors.

Prepare a fresh solution monthly.

5.8 Glassware

5.8.1 Volumetric flasks 100, 50, 25, 10 and 5 ml (tolerance better than $\pm 0,4\%$)

5.8.2 Syringes and pipettes:

500 µl $\pm$ 5 µl syringe or pipette,

1 µl and 5 µl gas chromatography precision syringes.

5.8.3 Bulb pipettes (volumetric) 1, 2 and 5 ml class A

5.9 Columns and accessories for sample preparation

5.9.1 Commercial or self-packed solid-phase extraction columns:

3 ml silica gel column, adsorbent weight 500 mg, particle size 40 µm,

3 ml benzenesulphonic acid column, adsorbent weight 500 mg, particle size 40 µm.

5.9.2 Column adapter, for joining two columns

5.9.3 Vacuum manifold column processor – optional

6 Apparatus

6.1 Gas chromatograph (GC)

A high-resolution gas chromatograph with accurately reproducible oven temperature control, capable, when used with the appropriate column and conditions, of resolving the test mixture (5.5) at least as well as in figure A.1 (90 peaks observed) and of reproducing relative retention times to within $\pm 0,0015$.

The gas lines (carrier gas and make-up gas) shall be fitted with water vapour and oxygen traps.

The carrier gas supply system shall be capable of running with a 50 m column at maximum efficiency using He or H₂ carrier gas, e.g. adequate column head pressure.

The oven temperature programmer shall have a range that can be set to attain the required resolution.

6.1.1 Injector

Either an “on-column” injector or a “split/splitless” injector may be used.

6.1.2 Columns

Either a cross-linked 5 % phenyl-methyl silicone, stationary phase coated onto fused silica capillary column or a similar chemically bonded phase column. Their dimensions shall be as follows:

length	=	50 m to 60 m;
internal diameter	=	0,2 mm to 0,35 mm;
film thickness	=	0,1 μm to 0,25 μm .

NOTE – For suitable columns and manufacturers see B.2.

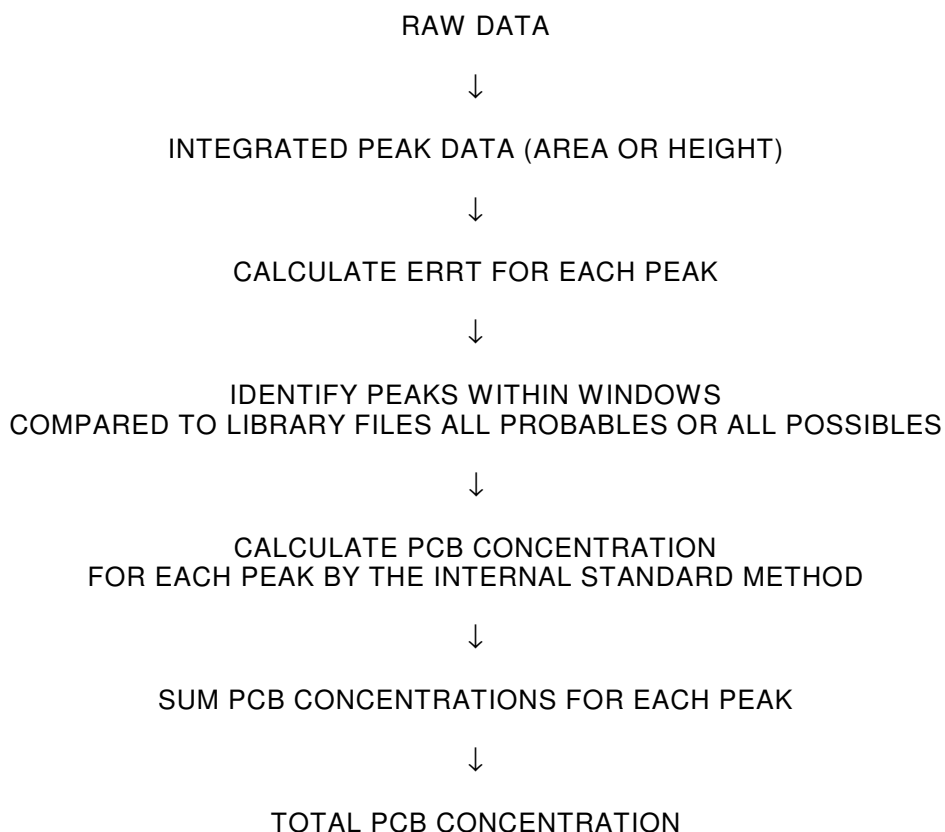
6.1.3 Detector

High-temperature Ni 63 electron capture detector (ECD) with adequate sensitivity to give a signal-to-noise ratio greater than 20 for one picogram of hexachlorobenzene (5.1.2) injected into the column.

The detector shall be operated within its linear range.

6.2 Data-processing system

Any data-processing system may be used with suitable software that can be programmed to process the operations shown below.



7 Sample

7.1 Sampling

Sampling shall be made in accordance with procedures described in IEC 60475.

To avoid cross-contamination of the samples, it is recommended that all the auxiliary material used (tubing, fittings, corks, connections, etc.) be disposable (single-use) and free from interfering compounds.

7.2 Sample preparation

Only glass or metal apparatus is suitable for sample preparation and the determination except for disposable pipette tips and columns made of plastic. All equipment shall be free from PCBs and interfering substances.

If the samples have a free-water phase, it shall be separated from the oil phase prior to further analysis, for example by centrifuging. Emulsified water perceived as opacity can be removed by adding sodium sulfate in portions and shaking until a clear sample is obtained.

The sample should be homogenized, e.g. by shaking the sample by hand for 3 min. An ultrasonic bath may also be used for this purpose.

8 Chromatograph operating conditions

8.1 General

The operating conditions given below have been found to be adequate but they should be optimized with each GC system so that gas chromatograms similar to the one shown in annex A can be obtained from dilutions of the test mixture in 5.5. Using hydrogen as carrier gas, a satisfactory separation will be obtained in 30-40 min (figure A.1). With helium carrier gas, the separation will take longer, 55-60 min.

8.2 Injectors

Set up the injector according to the manufacturer's instructions. Typical settings for this analysis are as follows:

Split/splitless injector

Splitless mode: T = 240 °C to 280 °C

Split mode: T = 250 °C to 280 °C, split ratio = 5:1 to 50:1

On-column injector: T = 50 °C to 110 °C according to the solvent used.

8.3 Oven temperature program

Injector mode	Split	Splitless	On-column
Initial isothermal period	0 – 2 min	1 min	0,5 min
Initial temperature	130 °C	50 °C	70 °C
Temperature program	130 °C to 290 °C at 2,5 °C/min	50 °C to 130 °C at 40 °C/min 130 °C to 290 °C at 2,5 °C/min	70 °C to 130 °C at 40 °C/min 130 °C to 290 °C at 2,5 °C/min
Rest time to final temperature	5 min	5 min	5 min
Cool down to	130 °C	50 °C	70 °C

8.4 Carrier gas flow rate

Adjust inlet pressure to give a flow rate through the column of 1 ml/min at 130 °C, e.g. 270 kPa for He.

NOTE – Hydrogen carrier gas is effective in reducing column pressure head and analysis time

8.5 Electron capture detector (ECD) settings

Temperature: 300 °C to 350 °C.

Electrical control: use manufacturer's recommended settings to give the best conditions for linearity of the detector.

Make-up gas, flow rate: 20 ml/min to 50 ml/min, according to manufacturer's recommendations.

9 Data-processing system

The system should be prepared in readiness for the start according to the manufacturer's instructions. Most systems require designation of a minimum of two reference points including the internal standard DCB.

9.1 Data files

The method requires data files containing experimental data (ERRT) and data originating from the literature. For each peak of single or coeluting congeners the following data is filed in order of increasing ERRT (see table A.1):

experimental relative retention time (ERRT);

congener numbers;

relative response factors (RRFs).

Two sets of RRFs based on data originating from [4]* are provided in table A.1. A weighted average response factor was calculated for each peak containing coeluting congeners using the relative proportions of the congeners found in commercial mixtures using data from [5], [6] and [7].

"All probable"

Some congeners have never been observed in commercial PCB mixtures. So, in those cases where more than one congener co-elutes under one chromatogram peak, the RRF of the group of congeners is weighted by exclusion of congeners not found in commercial mixtures. Use this data set with unknowns and mixtures of commercial products.

"All possible"

This class includes all 209 PCB congeners. This data set is included for use with dechlorinated materials.

Table A.1 shows that where there is no co-elution (for example peak n° 48) the RRF of each set has the same value and where there is co-elution (for example peak n° 49) there are different values for the different sets.

RRFs in table A.1 are corrected for the instrument being used by the calibration procedure in clause 11.

* Figures in square brackets refer to the bibliography given in annex C.

9.2 Co-eluting congeners

More than one congener may co-elute under a single peak, the programme should group peaks together if they fall within the window of $\pm 0,0015$ from the RRT. See table A.2 for individual congener RRTs and elution order.

10 Checks of instrumental performance

When initially implementing this method and after major repairs and replacement of critical instrumentation components (specifically EC detector and GC column), each laboratory that uses this method shall operate a performance control programme. This should include verification of sensitivity, resolution and linearity range. It is recommended to monitor the performance routinely at appropriate time intervals.

10.1 Sensitivity check

The ECD shall have sufficient sensitivity to give a signal-to-noise ratio (S/N) greater than 20 for one picogram of hexachlorobenzene injected into the column.

10.2 Linearity check

The response of the electron capture detector is proportional to the quantity of PCBs injected only within a limited range; if the quantities of PCBs passing through the detector become excessive, the response will cease to be linear. Determine the linear range as follows:

10.2.1 Use the stock solution of selected PCB congeners (5.6). Dilute the solution with solvent (5.1.1) containing 100 mg/ml of insulating liquid (5.1.3) in suitable steps e.g. 1, 2, 5, 20, 50, 100 dilution. Submit 500 μ l of each solution to the clean-up procedure (11.1.3) and add 10 μ l of C30 solution (5.1.4) to the 5 ml collecting flask before making up to the mark with solvent. The final dilutions are now, e.g. 10, 20, 50, 200, 500, 1 000. Each solution contains 20 ng/ml C30 and the eluate from 10 mg/ml insulating liquid. Inject a suitable quantity (the same each time) into the GC according to the injection system using the chromatographic conditions in clause 8.

10.2.2 The use of congeners 31, 118 and 180 that are present in major proportions in commercial mixtures plus the internal standard C209 (DCB) is recommended.

Measure the peak area or height (R_j) for the specified congeners 31, 118, 180 and 209 and calculate the concentration (B_j) of each congener in ng/ml for each dilution.

Use the area or height of the C30 peak to check that the correct volume has been injected. The area/height of the C30 peak for the series of injections shall not vary by more than ± 5 % of the average for that series of injections. Tests that fall outside of this range shall be repeated.

Calculate the sensitivity factor S_j for each congener and each dilution:

$$S_j = \frac{R_j}{B_j}$$

Plot S_j versus B_j (see figure 1).

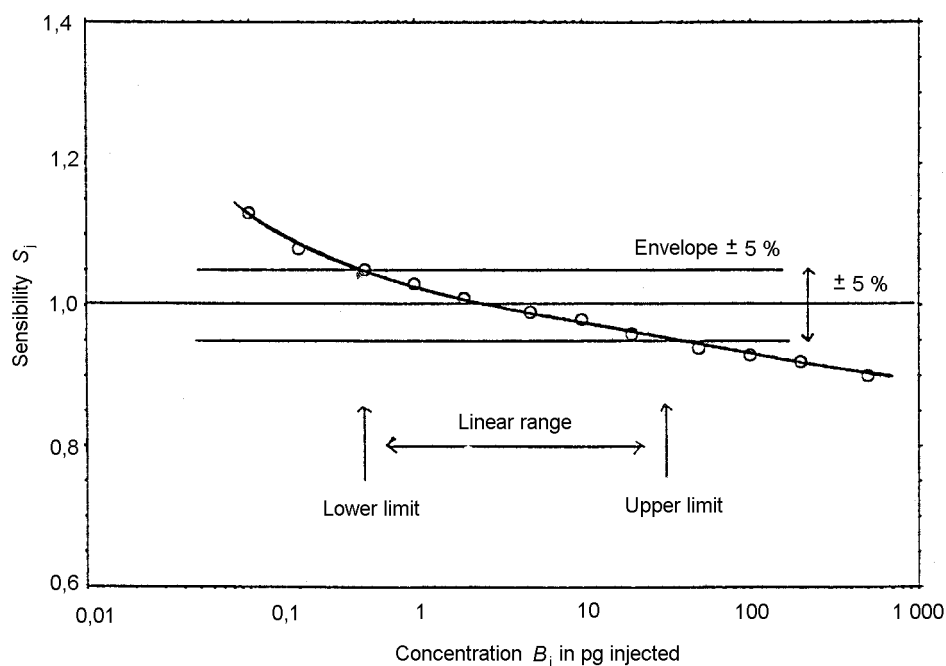


Figure 1 – Linearity check

10.2.3 The linearity plot is a line through the data points (figure 1). This line must be within 5 % of the constant value obtained by a least square fit. The upper limit of the linear range is the point where the plot crosses the – 5 % envelope and the lower limit level is where the line crosses the + 5 % envelope. The linear range of the detector is defined in figure 1.

10.2.4 Relating the linear range of the ECD to amounts of commercial mixtures. The maximum amounts of commercial mixture that may be injected to ensure they fall within the linear range of the detector may be calculated from the respective congener (see table 1).

Table 1 – Typical amounts of the major congeners in Aroclor® mixtures

Aroclor® solutions	Aroclor® concentration ng/ml	Congener number	Congener concentration ng/ml
1242	500	31	23
1254	500	118	32
1260	500	180	36
NOTE – The concentrations are approximate; a solution containing 500 ng/ml of Aroclor® is a 1 in 100 solution of a 50 mg/l Aroclor® standard.			

10.3 Resolution check

Treat 500 µl of solution (5.5) as per 11.1.3. Using optimised chromatographic parameters, inject a suitable aliquot in the linear range of the ECD.

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Calculate the resolution (R) for the pairs of congeners C28/C31 and C141/C179 (identified as in figure A.1). Resolution R is expressed as the ratio of the distance between the maxima of the peaks to the average of their peaks width at the base using the formula:

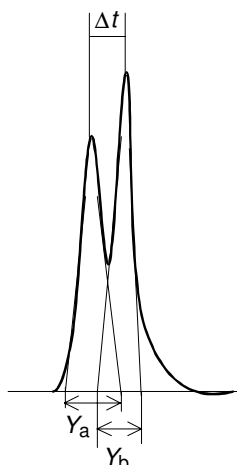
$$R = 2 \Delta t / (y_a + y_b)$$

The resolution shall be at least:

28/31 0,5

141/179 0,8

$$R = \frac{2 \Delta t}{y_a + y_b}$$



Providing the resolution is satisfactory, this chromatogram can be used for the determination of ERRTs (11.4).

11 Procedure

Safety precautions

Exercise normal laboratory safety precautions, wear gloves, impervious to mineral oil and light hydrocarbon solvents. Use only small quantities of flammable solvents on the workbench; handle larger volumes in a fume cupboard.

Ensure proper handling and disposal of PCB's and PCB contaminated equipment according to local regulations.

11.1 *Sample treatment (clean-up)*

11.1.1 *Test portion*

Weigh to the nearest 0,001 g, 0,9 g to 1,0 g of the test sample into a 10 ml volumetric flask. Add 1 ml, by pipette (5.8.3) of internal standard solution IS 2 (5.4.1) or IS 0,5 (5.4.2). Make up to the mark with solvent (5.1.1). Shake well to mix. If the sample is obviously wet, shown by the opacity of the solution, add anhydrous sodium sulphate and shake until a clear solution is obtained. This solution is designated solution A.

NOTE – For samples of unknown PCB content, use IS 2 (5.4.1). For better accuracy, IS 0,5 is preferred for samples with expected PCB content less than 20 mg/kg.

11.1.2 *Solid-phase column preparation*

Attach the sulphonic acid column (5.9.1) to the top of the silica gel column (5.9.1) using an adapter (5.9.2).

Elute the column assembly three times with 2 ml of the solvent (5.1.1) in order to purify the stationary phase. Do not allow the adsorbent to dry at this stage.

11.1.3 *Clean-up procedure*

Transfer 500 µl ± 5 µl of solution A (11.1.1) onto the top of the sulphonic acid upper column.

Add 0,5 ml solvent and apply a slight vacuum to distribute the sample evenly over the packing of the upper column. Wait at least 30 s before elution.

The elution shall be carried out at a maximum flow rate of 2 ml/min and the columns eluted each time until the solvent level is just above the top of the adsorbent (except for the final elution).

Elute the column combination twice with 1 ml aliquots of solvent (5.1.1) collecting the eluate in a 5 ml volumetric flask. Remove the sulphonic acid upper column and the adapter and elute the silica gel column twice with 0,5 ml of solvent, collecting the eluate in the same volumetric flask. Dilute to the volume mark with solvent (5.1.1); shake well to mix. This solution is designated solution B and used for GC analysis.

For some samples additional clean-up techniques may be necessary (see B.5).

11.1.4 *Recovery*

Dilute an Aroclor® solution (5.2.1) in solvent (5.1.1) to obtain a concentration of 5 mg/l.

This solution is designated solution C.

Run a 500 µl aliquot of solution C through the clean-up procedure (11.1.3). The 5 ml eluate obtained is designated solution D.

Dilute 500 µl of solution C to 5 ml with solvent (5.1.1) to give a concentration of 0,5 mg/l Aroclor®. This solution is designated solution E.

Add 50 µl internal standard solution IS2 (5.4.1) to solution D (from clean-up) and to solution E.

Inject solutions D and E into the GC and obtain a chromatogram and area table for each solution. Calculate the total amount of PCB for the Aroclor® in each solution using the internal standard method (11.7.2.2) and calculate the recovery as follows:

$$\% \text{ recovery} = \frac{\text{Total PCB content (mg) solution D}}{\text{Total PCB content (mg) solution E}} \times 100$$

The calculated recovery shall be greater than 95 %.

11.1.5 *Commercial PCB standards*

50 mg/kg commercial mixtures of PCBs in oil are used (5.2.2). If a standard of lower concentration is required, this is prepared by diluting (by weight) with unused PCB-free insulating liquid.

Commercial PCB standards are treated the same as the samples (11.1) and submitted to the clean-up procedure.

11.2 *Background check*

Run each new batch of solvent and a blank prepared with PCB-free insulating liquid (5.1.3) as per 11.1 through the GC to ensure that there are no spurious peaks.

A blank test portion should be run with every batch of samples and at least every 20 samples.

11.3 *Determination*

11.3.1 Sample and commercial PCB standard solutions, solution B (11.1.3) are injected into the GC. The GC is run under optimized conditions (clause 8).

11.3.2 Commercial PCB standards are run with every batch of samples, at least once every 10 samples. Choose standards appropriate to the samples; typically run 10 mg/kg and 50 mg/kg standards of Aroclor® 1260 in oil, (this is the most common commercial product) and other standards as required. Blank and control samples shall be subsequently used for quality assurance purposes.

NOTE – Chromatograms of all commercial materials likely to be encountered may be run and retained for type recognition purposes.

11.3.3 Integrate the chromatogram to obtain a peak table listing peak number, retention time, peak area and/or peak height, as would be produced by any GC integrating system.

11.4 *Determination of experimental relative retention times (ERRT)*

11.4.1 Run the test mixture (5.5) prepared as per 11.1, using the same GC conditions that are used for sample analyses. Identify all the peaks by comparison with the example in figure A.1 and calculate the ERRT for each peak as follows:

$$ERRT_x = \frac{t_x - t_{30}}{t_{209} - t_{30}}$$

where

x is the chosen peak;

30 is the congener 30 (reference);

209 is the congener 209 (reference and internal standard);

t is the retention time from injection.

Tabulate the results in a similar manner to the example as shown in table A.1 and enter the results in the data files (clause 9).

NOTES

- 1 ERRTs shall be determined and entered into the data files for each individual GC system. The system should be recalibrated if there are any changes in GC conditions (e.g. temperature program, etc.).
- 2 Congeners 30 and 209 are chosen as reference peaks for the determination of ERRT as they are at each end of the chromatogram (test mixture) isolated from congeners occurring in commercial mixtures and enable accurate repeatable values of ERRTs to be obtained.

11.4.2 Reference peaks for data-processing system

Use the ERRT values of the congeners listed in table 2 below (determined in 11.4.1) to designate the reference peaks in the data-processing system.

Table 2 – Reference peaks/congeners

Peak numbers	Reference peak congener numbers	ERRT (examples)
9	30	0,000
33	44	0,225
46	56/60	0,342
57	77/110	0,427
74	138/160/163	0,574
90	180	0,703
105	209	1,000
NOTE – With the exception of C30 and C209, the reference peaks were chosen because they are major components of commercial mixtures. They will not occur in all samples, the number of reference peaks found will depend on the PCB mixture.		

11.5 Calculation of corrected relative response factors (CRRF)

11.5.1 The experimental relative response factors (ERRFs) of the congeners may vary from instrument to instrument depending on the injection mode and the conditions of the electron capture detector. They may also differ from those originating from [4].

11.5.2 The chromatogram is divided into nine segments (see figure A.1) each represented by one of the congeners listed in table 3. These congeners are normally present in commercial mixtures as major components (see table A.1).

11.5.3 Using optimised chromatographic conditions as in clause 8, inject a suitable aliquot of the cleaned-up congener mix calibration solution (5.7).

Determine the relative response factors (to DCB) for the congeners listed in table 3 using the following equation:

$$ERRF_i = \frac{A_i \times M_s}{A_s \times M_i}$$

where

A_s is the peak area/height for internal standard (DCB);

A_i is the peak area/height for congener i;

M_s is the concentration (µg/ml) of internal standard (DCB);

M_i is the concentration (µg/ml) of congener i.

NOTE – CAUTION: some software packages may produce the inverse of this $ERRF_i$.

Take the average ERRF for a minimum of three determinations

Table 3 – Congeners for ERRF calibration

Peak number	Segment number	Congener number (IUPAC)	ERRT (examples)	RRF (table A.2)
12	1	18	0,028	0,275
22	2	31	0,114	0,493
33	3	44	0,225	0,460
49	4	101	0,356	0,587
63	5	118	0,477	0,764
74	6	138	0,574	0,726
90	7	180	0,703	1,137
95	8	170	0,759	0,659
102	9	194	0,877	1,640

11.5.4 From the determined ERRF and the theoretical RRF value given in table 3, calculate the correction factor K_i for each congener:

$$K_i = \text{ERRF}/\text{RRF}$$

for example, for congener 180: segment 7

where

determined ERRF = 1,030

literature theoretical RRF = 1,137 (table A.2)

$$K_{180} = \frac{1,030}{1,137} = 0,906$$

11.5.5 Multiply the RRFs of all peaks in each segment of the chromatogram (table A.1) by the correction factor calculated for the corresponding selected congener.

Example: segment 7: congener 180;
multiply each RRF by 0,906.

The resulting table of the corrected relative response factors (CRRF) for possibles and probables will be the one to use for data files (9.1).

11.6 Examination of the chromatograms

Chromatograms should be examined visually for any spurious peaks or chromatography problems and for interferences.

NOTE – The commercial mixtures of "tetrachlorobenzyl toluene" may be encountered and can be mistaken for PCBs. These can be identified by their distinctive patterns in the PCB region of the chromatogram.

11.7 Calculation of results

11.7.1 Qualitative analysis

Compare the sample chromatograms with standard chromatograms to identify commercial materials such as Aroclor® 1242, 1254 and 1260 (see figure B.1).

11.7.2 Quantitative analysis

11.7.2.1 Calculation

The computer programme/software should produce a list, containing peak number with corresponding PCB congener numbers and the concentration of PCBs (mg/l) for each peak calculated from the concentration of internal standard DCB.

11.7.2.2 Mass of PCB in each peak

The mass of each congener or co-eluting congeners (for each peak i) is calculated by the internal standard method using the corrected relative response factors. Use the corrected response factors derived from the "all probable" RRF column in table A.1 where there is no evidence for dechlorination or the "all possible" values for dechlorinated samples (see 9.1).

$$m_i \text{ (mg)} = \frac{m_s \times A_i \times \text{RRF}_s}{A_s \times \text{CRRF}_i}$$

where:

i represents the peak due to a congener or co-eluting congeners;

m_i is the mass of peak i in the test portion in milligrams;

m_s is the mass of internal standard, in milligrams in the test portion 11.1.1 (nominally 0,002 or 0,0005);

A_i is the the area/height of peak i ;

A_s is the area/height of internal standard peak;

RRF_s is the relative response factor of internal standard (= 1,000);

CRRF_i is the corrected relative response factor of peak i .

11.7.2.3 The mass of all the individual peaks are summed to give the total mass of PCBs (mg) in solution B.

Total PCB content of the sample (mg/kg) is calculated using the initial weight of sample in the test portion (11.1.1). These values may be entered into the software so that the actual result is the total PCB content of the sample in mg/kg.

$$\text{Concentration of PCBs in sample (mg/kg)} = \frac{\sum m_i \times 1000}{\text{weight of sample (g)}}$$

12 Test report

Report as total PCB content in mg/kg to the nearest 1 mg/kg.

State the data set that was used, e.g. "all probables".

13 Detection limit

Detection limit depends on several factors such as injection volume, injection mode, condition of the detector, etc. For a single peak the detection limit is about 0,1 mg/kg. It has been evaluated that quantification in terms of total PCB's is reliable only above 2 mg/kg.

14 Precision

14.1 *Repeatability*

Duplicate determinations carried out by one operator should be considered suspect at the 95 % confidence level if they differ by more than $2 + 0,1 x$ (where x is the average of the duplicate determinations).

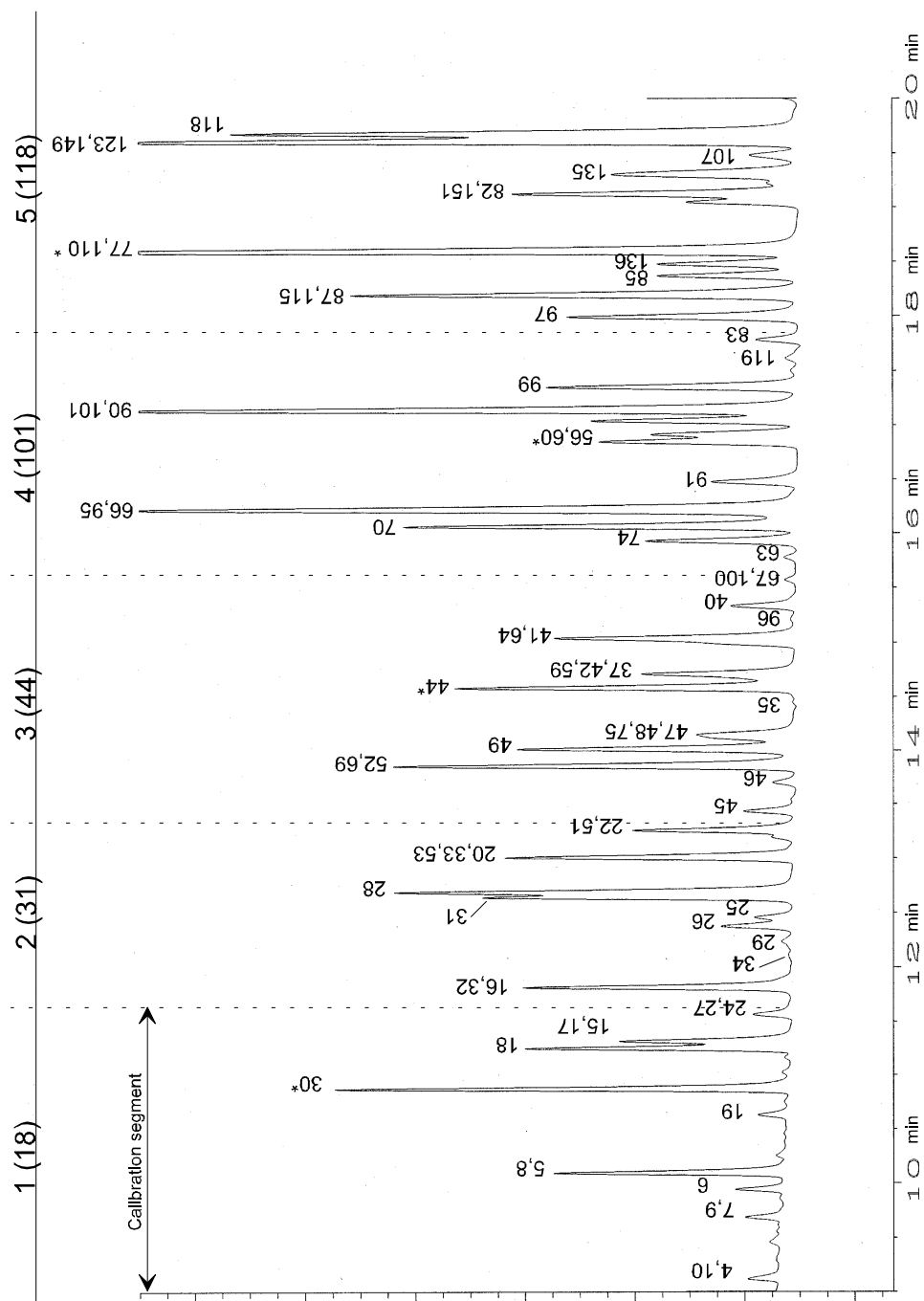
14.2 *Reproducibility*

When two laboratories carry out tests on identical test material, each shall produce duplicate results and report their average.

The two averages should be considered suspect at the 95 % confidence level if they differ by more than $2 + 0,25 x$ (where x is the average of the two averages).

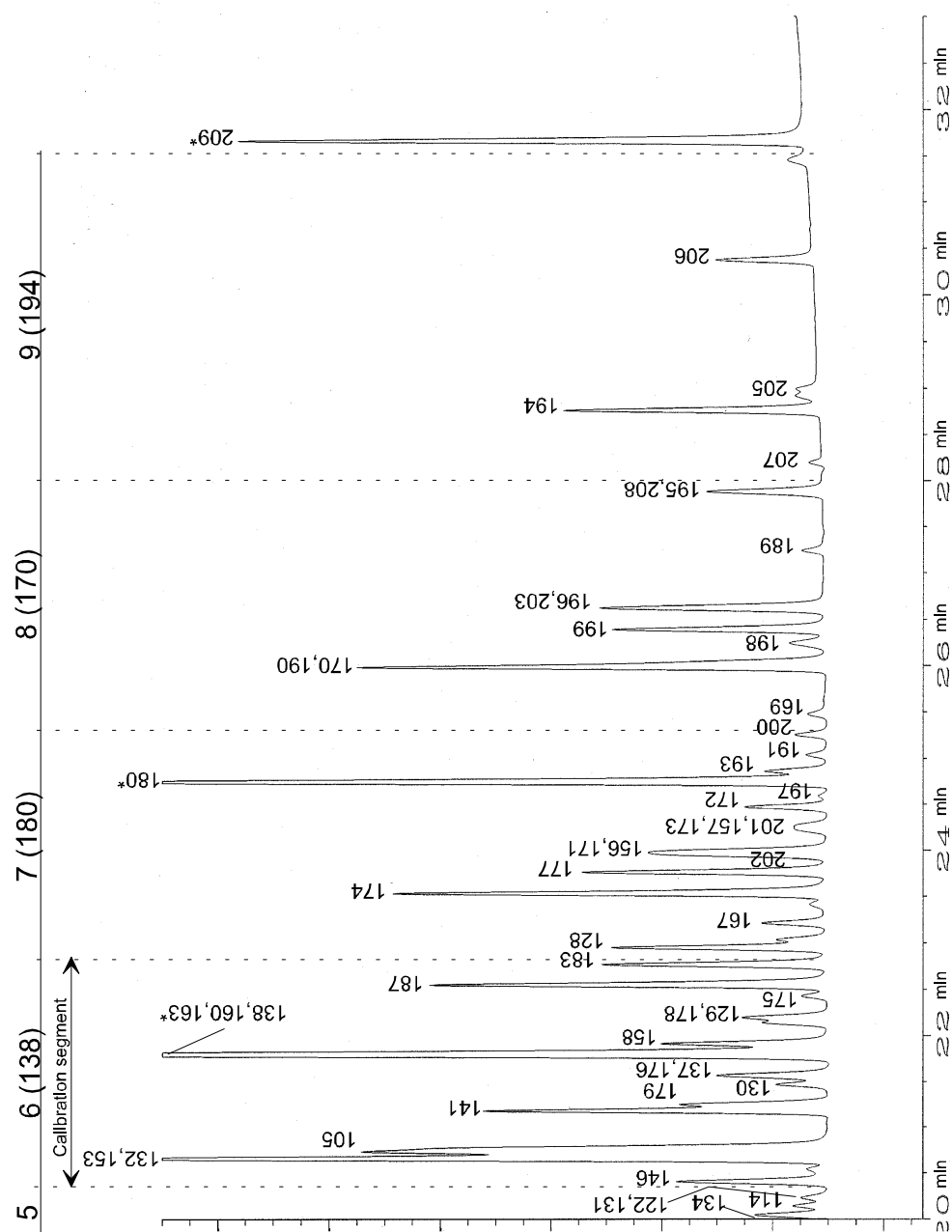
Annex A (normative)

Test mixture



* Reference peaks, see 11.4.2.
Congeners, see 11.5.3.

Figure A.1 – Chromatogram for test mixture 1242/1254/1260 (5.5)



* Reference peaks, see 11.4.2.
Congeners, see 11.5.3.

Figure A.1 (concluded)

Table A.1 – List of congeners, retention times and relative response factors

Calibration	Segment		Congener numbers		RRFs (to DCB)	
Peak number		ERRT (example)	All probables (IUPAC numbers)	All possibles (IUPAC numbers)	All probables	All possibles
1	1	(-0,223)	–	1		0,035
2		(-0,127)	2,3	2,3	0,026	0,026
3		-0,124	4,10	4,10	0,217	0,131
4		-0,082	7,9	7,9	0,453	0,473
5		-0,062	6	6	0,334	0,334
6		-0,052	5,8	5,8	0,105	0,143
7	1	(-0,032)	–	14	–	0,268
8		-0,014	19	19	0,267	0,267
9		0,000	–	30	–	0,720
10		(0,006)	–	11	–	0,039
11		(0,013)	–	12,13	–	0,166
12		0,028	18	18	0,275	0,275
13		0,032	15,17	15,17	0,182	0,182
14	1	0,048	24,27	24,27	0,541	0,565
15	2	0,064	16,32	16,32	0,346	0,318
16		(0,072)	–	23	–	0,439
17		0,082	34	34,54	0,535	0,427
18		0,089	29	29	0,557	0,557
19		0,099	26	26	0,529	0,529
20	2	0,102	25	25	0,439	0,439
21		(0,110)	–	50	–	0,599
22		0,114	31	31	0,493	0,493
23		0,117	28	28	0,750	0,750
24		0,136	20,33,53	20,21,33,53	0,405	0,569
25	2	0,151	22,51	22,51	0,936	0,960
26	3	0,159	45	45	0,474	0,474
27		0,165	–	36	–	0,459
28		0,177	46	46	0,411	0,411
29		0,185	52,69	39,52,69,73	0,389	0,473
30		0,193	49	38,43,49	0,569	0,474
31		0,199	47,48,75	47,48,62,65,75	0,621	0,709
32	3	0,216	35	35,104	0,329	0,365
33		0,225	44	44	0,460	0,460
34		0,232	37,42,59	37,42,59	0,613	0,577
35		0,241	71,72	71,72	–	0,448
36		0,249	41,64	41,64	0,507	0,510
37		0,255	96	68,96	–	0,508
38		0,266	40	40,57,103	0,634	0,565
39	3	0,283	67,100	67,100	0,524	0,521
40	4	0,287	63	58,63	0,639	0,587
41		0,294	74	61,74,94	0,589	0,686
42		0,302	70	70,76,98	0,578	0,545
43		0,310	66,95	66,80,88,93,95	0,417	0,531
44		(0,322)	121	102	–	0,672
45		0,323	91	55,91	0,501	0,615
46		0,342	56,60	56,60,155	0,801	0,712
47		0,346	92	92	0,472	0,472
48		0,353	84	84	0,339	0,339
49	4	0,356	90,101	89,90,101	0,581	0,538

Table A.1 (continued)

Calibration	Segment		Congener numbers		RRFs (to DCB)	
Peak number		ERRT (example)	All probables (IUPAC numbers)	All possibles (IUPAC numbers)	All probables	All possibles
50	4	0,366	99	79, 99, 113	0,528	0,614
51		0,378	119	112,119,150	0,723	0,650
52	4	0,388	83	78,83,109	0,557	0,665
53	5	0,398	97	86,97,152	0,554	0,571
54		0,408	87,115	81,87,111,115, 116,117,125, 145	0,903	0,774
55		0,416	85	85	0,649	0,649
56		0,422	136	120,136,148	0,398	0,510
57		0,427	77,110	77,110	0,559	0,453
58		(0,448)	—	154	—	0,500
59	5	0,451	151,82	151,82	0,681	0,681
60		0,460	135	124,135,144	0,617	0,710
61		0,468	107	107,108,147	0,718	0,727
62		0,474	123,149	106,123,149	0,511	0,656
63		0,477	118	118,139,140	0,764	0,663
64		0,495	134	134,143	0,644	0,633
65		0,499	114	114	0,901	0,901
66	5	0,503	122,131	122,131,133	0,662	0,862
67	6	0,510	146	146,161,165 188	0,639	0,770
68		0,521	132,153	132,153,184	0,615	0,709
69		0,528	105	105,127,168	0,825	0,690
70		0,546	141	141	1,187	1,187
71		0,550	179	179	0,723	0,723
72		0,559	130	130	0,836	0,836
73		0,564	137,176	137,176	0,939	0,953
74	6	0,574	138,160,163	138,160,163, 164	0,771	0,878
75		0,579	158	158,186	0,994	1,034
76		0,590	126,129,178	126,129,178	0,670	0,919
77		0,601	175	166,175	0,335	0,625
78		0,607	187	159,182,187	0,985	0,949
79	6	0,616	183	162,183	0,857	0,882
80	7	0,627	128	128	1,043	1,043
81		0,636	167	167	0,936	0,936
82		(0,641)	185	185	1,262	1,262
83		0,652	174	174,181	0,708	1,058
84		0,662	177	177	0,886	0,886
85		0,670	202	202	1,023	1,023
86		0,671	156,171	156,171	1,124	1,124
87	7	0,683	201*,157,173	201*,157,173	0,662	0,662
88		0,691	172	172,204	1,029	0,867
89		0,696	197	192,197	1,009	1,090
90		0,703	180	180	1,137	1,137
91		0,708	193	193	1,244	1,244
92		0,716	191	191	1,294	1,294
93	7	0,727	200*	200*	1,010	1,010

Table A.1 (concluded)

Calibration	Segment		Congener numbers		RRFs (to DCB)	
Peak number		ERRT (example)	All probables (IUPAC numbers)	All possibles (IUPAC numbers)	All probables	All possibles
94	8	0,736	169	169	0,734	0,734
95		0,759	170,190	170,190	0,782	0,904
96		0,769	198	198	0,939	0,939
97	8	0,775	199*	199*	0,705	0,705
98		0,785	196,203	196,203	1,287	1,265
99		0,812	189	189	1,325	1,325
100	8	0,838	195,208	195,208	0,593	0,593
101	9	0,852	207	207	1,164	1,164
102		0,877	194	194	1,640	1,640
103		0,885	205	205	1,234	1,234
104	9	0,945	206	206	1,469	1,469
105		1,000	209 (internal standard)	209 (internal standard)	1,000	1,000
* Numbered according to IUPAC rules. Ballschmitter & Zell numbering [6] as follows: 199 (IUPAC) = 201 (Ballschmitter) 200 (IUPAC) = 199 (Ballschmitter) 201 (IUPAC) = 200 (Ballschmitter) NOTES 1 The ERRT values in the table are examples determined from the chromatogram in figure A.1 and should not be used in the method. ERRT values shall be determined for the individual GC system being used (11.4.1). 2 Values appearing in parentheses are calculated values for peaks (congeners) that do not appear in the test mixture (5.5), e.g. ERRT (Peak 44 = 0,322). 3 References [3], [4], [5], [6] and [7], were used to aid the identification of congeners represented by each peak in the test mixture, listed in this table and shown on the chromatogram in figure A.1.						

Table A.2 – Elution order of PCB

Congener number (IUPAC)	Relative retention time to DCB	Relative factor response	Congener number (IUPAC)	Relative retention time to DCB	Relative response factor
1	0,147	0,035	48	0,443	0,488
2	0,185	0,035	65	0,445	0,738
3	0,188	0,017	62	0,446	1,008
10	0,214	0,230	35	0,451	0,329
4	0,214	0,033	104	0,453	0,400
7	0,245	0,606	44	0,460	0,460
9	0,245	0,341	37	0,463	0,509
6	0,258	0,334	59	0,463	0,527
8	0,265	0,181	42	0,464	0,695
5	0,265	0,105	72	0,475	0,484
14	0,283	0,268	71	0,475	0,411
19	0,290	0,267	41	0,475	0,480
30	0,302	0,720	64	0,476	0,539
11	0,309	0,394	68	0,480	0,637
12	0,314	0,157	96	0,482	0,378
13	0,316	0,176	40	0,486	0,634
18	0,322	0,275	103	0,490	0,533
15	0,323	0,094	57	0,491	0,527
17	0,324	0,362	100	0,497	0,515
24	0,334	0,696	67	0,497	0,527
27	0,336	0,435	58	0,502	0,535
16	0,345	0,392	63	0,504	0,639
32	0,346	0,244	61	0,508	1,074
23	0,359	0,439	94	0,508	0,396
34	0,360	0,535	74	0,509	0,589
54	0,362	0,320	70	0,515	0,578
29	0,364	0,556	76	0,515	0,509
26	0,373	0,529	98	0,516	0,548
25	0,375	0,439	102	0,517	0,400
50	0,382	0,599	93	0,518	0,586
31	0,383	0,493	66	0,519	0,567
28	0,384	0,750	80	0,521	0,639
21	0,394	0,931	95	0,521	0,389
33	0,397	0,392	88	0,523	0,605
20	0,397	0,636	121	0,526	0,672
53	0,399	0,317	91	0,529	0,501
51	0,404	0,527	55	0,530	0,728
22	0,406	0,960	155	0,540	0,515
45	0,413	0,474	56	0,541	0,728
36	0,417	0,259	60	0,541	0,892
46	0,424	0,411	92	0,547	0,472
39	0,428	0,305	84	0,547	0,339
69	0,430	0,705	89	0,551	0,493
73	0,434	0,510	90	0,554	0,536
52	0,434	0,367	101	0,554	0,587
43	0,437	0,442	113	0,559	0,530
38	0,438	0,413	99	0,560	0,538
49	0,439	0,569	79	0,562	0,774
47	0,442	0,745	119	0,569	0,723
75	0,442	0,567	150	0,569	0,498

Table A.2 (concluded)

Congener number (IUPAC)	Relative retention time to DCB	Relative factor response	Congener number (IUPAC)	Relative retention time to DCB	Relative response factor
112	0,570	0,728	130	0,694	0,836
109	0,573	0,845	176	0,696	0,9230
78	0,574	0,979	137	0,698	0,976
83	0,574	0,557	160	0,705	1,046
152	0,578	0,460	163	0,705	0,876
97	0,581	0,554	164	0,705	0,865
86	0,582	0,700	138	0,705	0,726
116	0,584	1,228	186	0,707	1,074
125	0,585	0,488	158	0,708	0,994
81	0,586	0,629	129	0,715	0,875
145	0,586	0,596	126	0,716	0,418
117	0,586	0,781	178	0,718	0,545
115	0,588	0,995	166	0,721	0,915
87	0,588	0,896	175	0,725	0,335
111	0,589	0,580	182	0,729	0,990
85	0,593	0,649	187	0,729	0,985
148	0,595	0,486	159	0,729	0,872
120	0,596	0,654	183	0,736	0,857
136	0,596	0,390	162	0,737	0,906
77	0,600	0,335	128	0,739	1,043
110	0,602	0,571	167	0,745	0,936
154	0,605	0,500	185	0,748	1,262
82	0,615	0,679	174	0,759	0,708
151	0,619	0,689	181	0,759	1,409
135	0,625	0,617	177	0,765	0,886
144	0,629	0,769	171	0,771	1,028
124	0,627	0,745	202	0,771	1,023
147	0,630	0,527	156	0,772	1,220
108	0,631	0,935	173	0,777	1,795
107	0,632	0,718	157	0,780	1,051
123	0,634	0,583	201	0,781	0,324
149	0,636	0,502	204	0,783	0,705
106	0,636	0,882	192	0,788	1,404
118	0,638	0,764	172	0,789	1,029
139	0,639	0,634	197	0,790	0,836
140	0,639	0,591	180	0,797	1,137
143	0,647	0,622	193	0,800	1,247
134	0,648	0,644	191	0,805	1,294
114	0,651	0,901	200	0,809	1,010
142	0,652	1,069	169	0,822	0,734
131	0,653	0,746	170	0,833	0,659
122	0,655	0,636	190	0,833	1,150
133	0,655	1,008	198	0,843	0,939
165	0,659	0,946	199	0,846	0,705
188	0,659	0,644	196	0,852	1,082
146	0,663	0,639	203	0,852	1,430
161	0,664	0,849	189	0,871	1,325
184	0,668	0,882	208	0,888	1,032
132	0,670	0,641	195	0,888	0,364
153	0,670	0,604	207	0,898	1,164
105	0,672	0,825	194	0,917	1,640
168	0,673	0,735	205	0,922	1,234
127	0,674	0,512	206	0,963	1,469
141	0,686	1,187	209	1,000	1,000
179	0,686	0,723			

NOTES

1 Results obtained with a high performance column: crosslinked 5 % phenyl-methyl silicone gum phase 50 m × 0,2 mm internal diameter x 0,11 µm film thickness.

2 The RRT's and RRF's in this table were calculated from the values in [4] to make them relative to DCB.

Annex B (informative)

General informations

Table B.1 – Systematic numbering of PCB-compounds

Number	Structure	Number	Structure	Number	Structure	Number	Structure
1	Monochlorobiphenyls	52	Tetrachlorobiphenyls	105	Pentachlorobiphenyls	161	Hexachlorobiphenyls
2	2	53	2,2',5,5'	106	2,3,3',4,4'	162	2,3,3',4,5',6
3	3	54	2,2',5,6'	107	2,3,3',4,5	163	2,3,3',4',5,5'
	4	55	2,2',6,6'	108	2,3,3',4',5	164	2,3,3',4',5,6
	Dichlorobiphenyls	56	2,3,3',4	109	2,3,3',4,5'	165	2,3,3',4',5',6
4	2,2'	57	2,3,3',4'	110	2,3,3',4,6	166	2,3,3',5,5',6
5	2,3	58	2,3,3',5	111	2,3,3',4,6	167	2,3,4,4',5,6
6	2,3'	59	2,3,3',5'	112	2,3,3',5,5'	168	2,3',4,4',5,5'
7	2,4	60	2,3,3',6	113	2,3,3',5,6	169	2,3',4,4',5',6
8	2,4'	61	2,3,4,4'	114	2,3,3'5',6		3,3',4,4',5,5'
9	2,5	62	2,3,4,5	115	2,3,4,4',5		Heptachlorobiphenyls
10	2,6	63	2,3,4,6	116	2,3,4,4',6	170	2,2',3,3',4,4',5
11	3,3'	64	2,3,4',5	117	2,3,4,5,6	171	2,2',3,3',4,4',6
12	3,4	65	2,3,4',6	118	2,3',4,4',5	172	2,2',3,3',4,5,5'
13	3,4'	66	2,3,5,6	119	2,3',4,4',6	173	2,2',3,3',4,5,6
14	3,5	67	2,3',4,4'	120	2,3',4,5,5'	174	2,2',3,3',4,5,6'
15	4,4'	68	2,3',4,5	121	2,3',4,5',6	175	2,2',3,3',4,5,6'
	Trichlorobiphenyls	69	2,3',4,5'	122	2',3,3',4,5	176	2,2',3,3',4,6,6'
16	2,2',3	70	2,3',4,6	123	2',3,4,4',5	177	2,2',3,3',4',5,6
17	2,2',4	71	2,3',4',5	124	2',3,4,5,5'	178	2,2',3,3',5,5',6
18	2,2',5	72	2,3',4',6	125	2',3,4,5,6'	179	2,2',3,3',5,6,6'
19	2,2',6	73	2,3',5,6	126	3,3',4,4',5	180	2,2',3,4,4',5,5'
20	2,3,3'	74	2,3',5',6	127	3,3',4,5,5'	181	2,2',3,4,4',5,6
21	2,3,4	75	2,4,4',5		Hexachlorobiphenyls	182	2,2',3,4,4',5,6'
22	2,3,4'	76	2,4,4',6	128	2,2',3,3',4,4'	183	2,2',3,4,4',5',6
23	2,3,5	77	2',3,4,5	129	2,2',3,3',4,4',5	184	2,2',3,4,4',6,6'
24	2,3,6	78	3,3',4,4'	130	2,2',3,3',4,5'	185	2,2',3,4,5,5',6
25	2,3',4	79	3,3',4,5	131	2,2',3,3',4,5'	186	2,2',3,4,5,6,6'
26	2,3',5	80	3,3',4,5'	132	2,2',3,3',4,6'	187	2,2',3,4',5,5',6
27	2,3',6	81	3,4,4',5	133	2,2',3,3',5,5'	188	2,2',3,4',5,6,6'
28	2,4,4'		Pentachlorobiphenyls	134	2,2',3,3',5,5'	189	2,3,3',4,4',5,5'
29	2,4,5	82	2,2',3,3',4	135	2,2',3,3',5,6'	190	2,3,3',4,4',5,6
30	2,4,6	83	2,2',3,3',5	136	2,2',3,3',6,6'	191	2,3,3',4,4',5',6
31	2,4',5	84	2,2',3,3',6	137	2,2',3,4,4',5	192	2,3,3',4,5,5',6
32	2,4',6	85	2,2',3,3',6'	138	2,2',3,4,4',5'	193	2,3,3',4',5,5',6
33	2',3,4	86	2,2',3,4,4'	139	2,2',3,4,4',6		Octachlorobiphenyls
34	2',3,5	87	2,2',3,4,5	140	2,2',3,4,4',6'	194	2,2',3,3',4,4',5,5'
35	3,3',4	88	2,2',3,4,5'	141	2,2',3,4,5,5'	195	2,2',3,3',4,4',5,6
36	3,3',5	89	2,2',3,4,6	142	2,2',3,4,5,6'	196	2,2',3,3',4,4',5,6'
37	3,4,4'	90	2,2',3,4,6'	143	2,2',3,4,5,6'	197	2,2',3,3',4,4',6,6'
38	3,4,5	91	2,2',3,4',5	144	2,2',3,4,5',6	198	2,2',3,3',4,5,5',6
39	3,4',5	92	2,2',3,4',6	145	2,2',3,4,5',6'	199	2,2',3,3',4,5,6,6'
	Tetrachlorobiphenyls	93	2,2',3,5,5'	146	2,2',3,4',5,5'	200	2,2',3,3',4,5',6,6'
40	2,2',3,3'	94	2,2',3,5,6'	147	2,2',3,4',5,6	201	2,2',3,3',4',5,5',6
41	2,2',3,4	95	2,2',3,5',6	148	2,2',3,4',5,6'	202	2,2',3,3',5,5',6,6'
42	2,2',3,4'	96	2,2',3,6,6'	149	2,2',3,4',6,6'	203	2,2',3,4,4',5,5',6
43	2,2',3,5	97	2,2',3',4,5	150	2,2',3,4',6,6'	204	2,2',3,4,4',5,6,6'
44	2,2',3,5'	98	2,2',3',4,6	151	2,2',3,5,5',6	205	2,3,3',4,4',5,5',6
45	2,2',3,6	99	2,2',3',4,6'	152	2,2',3,5,6,6'		Nonachlorobiphenyls
46	2,2',3,6'	100	2,2',4,4',5	153	2,2',4,4',5,5'	206	2,2',3,3',4,4',5,5',6
47	2,2',4,4'	101	2,2',4,4',6	154	2,2',4,4',5,6'	207	2,2',3,3',4,4',5,6,6'
48	2,2',4,5	102	2,2',4,5,5'	155	2,2',4,4',6,6'	208	2,2',3,3',4,5,5',6,6'
49	2,2',4,5'	103	2,2',4,5,6'	156	2,3,3',4,4',5		Decachlorobiphenyl
50	2,2',4,6	104	2,2',4,6,6'	157	2,3,3',4,4',5'	209	2,2',3,3',4,4',5,5',6,6'
51	2,2',4,6'			158	2,3,3',4,4',6		
				159	2,3,3',4,5,5'		
				160	2,3,3',4,5,6		

NOTES

- The number is used as a synonym for the corresponding PCB compound in tables and figures.
- This table is taken from [6] and shows Ballschmitter numbering.
- The numbers differ from the IUPAC rules as follows:
199 (Ballschmitter) = 200 (IUPAC)
200 (Ballschmitter) = 201 (IUPAC)
201 (Ballschmitter) = 199 (IUPAC)

B.1 Commercial PCBs*

<u>Producer</u>	<u>Country</u>	<u>Trade Name</u>
Monsanto	USA and Britain	Aroclor®
Bayer	Germany	Clophen®
Prodelec	France	Phenoclor® and Pyrалene®
Kanegafuchi	Japan	Kanechlor®
Mitsubishi	Japan	Santotherm®
Caffaro	Italy	Fenclor®/Apirolio®
Kenneclor	Japan	Kenneclor®
Sovol	Russian Federation	
Hevi-Duty Corp. Ferranti-Packard Ltd. Universal Mfg. Co.	USA	Askarel®

B.2 Suitable GC columns manufacturers*

<u>Column</u>	<u>Manufacturer</u>
Rt _x 5	Restek
DB5	J & W
SPB-5	Supelco
OV-5	Ohio Valley
HP-5, Ultra-Z	Hewlett Packard
RSL-200	Alltech
CP SIL 8CB	Chrompack
BP5	SGE
007-2	Quadrex
TRB-5	Tracer

B.3 Suppliers of calibration solution of PCB congeners*

B.3.1 National Research Council of Canada Institute for Marine Biosciences, Marine Analytical Chemistry Standards Program
1411 Oxford Street
Halifax Nova Scotia B3H3Z1
Canada

Standard CLB-1

Standard CLB-1 consists of a set of four solutions (A, B, C and D) containing 51 congeners.

Supplied by 1 ml ampoules with solutions containing 14-15 congeners each, all containing DCB (209). Concentrations are certified and values are given in the documentation supplied.

* This information is given for the convenience of users of this International Standard and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

IS 16082 : 2013
IEC 61619 : 1997

Only solutions CLB1-A and CLB1-D are used, they contain the following congeners:

CLB1-A: 18, 31, 40, 44, 49, 54, 77, 86, 87, 121, 153, 156, 159, 209

CLB1-D: 15, 101, 118, 138, 141, 151, 153, 170, 180, 187, 194, 195, 196, 199, 209

B.3.2 Supelco Switzerland
Chemin du Lavasson 2
CH-1196 Gland
Switzerland

Supelco PCB congeners mixture ref: 10156, 1 ml ampoule containing a solution of the following congeners at 10 µg/ml in hexane:

18, 31, 28, 20, 44, 52, 101, 105, 118, 138, 149, 153, 170, 180, 194, 209

Concentrations are certified and values are given in the documentation supplied.

B.3.3 Promochem
Postfach 1246
D-46469 Wessel
Germany

B.4 Commercial PCB standards*

These are available from various suppliers. Certified standards (Aroclors®) are available from the National Institute of Standards and Technology, US Department of Commerce, Standard Reference Materials, Building 202, Room 204, NIST, Gaithersburg, MD 20899.

* This information is given for the convenience of users of this International Standard and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

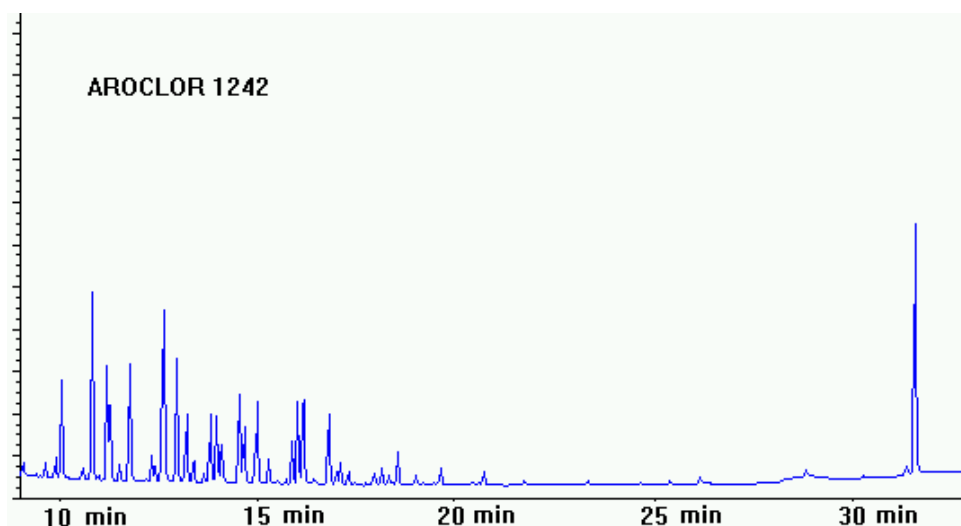


Figure B.1a

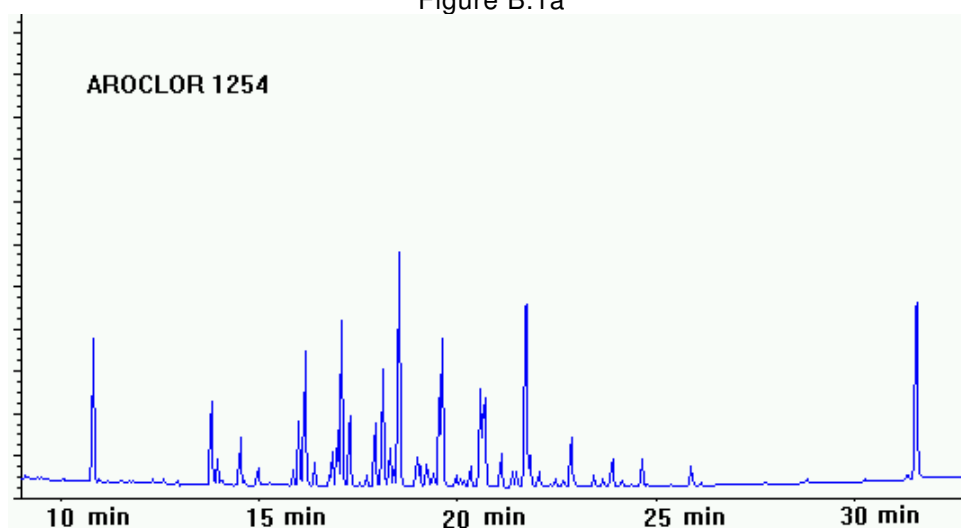


Figure B.1b

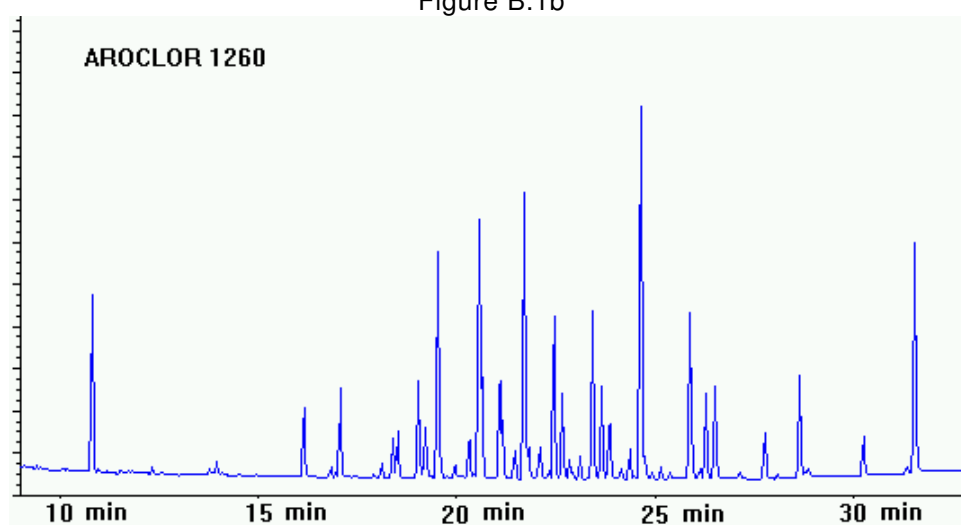


Figure B.1c

Figure B.1 – Examples of chromatograms obtained for the three main Aroclors®

B.5 Sample clean-up techniques

B.5.1 *Sulphuric acid treatment*

Transfer 2 ml $\pm$ 0,02 ml of solution A (11.1.1) into a 20 ml volumetric flask. Make up to the mark with solvent (5.1.1). Shake well to mix. This solution is designated solution B1. Transfer 10 ml of solution B1 into a convenient stoppered glass flask or other vessel. Carefully add 5 ml of concentrated sulphuric acid, stopper the vessel and shake vigorously at intervals for 5 min. Allow to separate completely (about 15 min).

If necessary transfer to a centrifuge tube and centrifuge to separate the phases. Take a portion of the upper phase for GC analysis.

B.5.2 *Benzenesulfonic acid/sulphuric acid column*

This clean-up is similar to the standard method clean-up (11.1) except that silica gel/sulphuric acid material is added to the top of the sulphonic acid column.

B.5.2.1 *Preparation of silica gel/sulphuric acid material*

Weigh 28 g $\pm$ 1 g of chromatographic grade activated silica gel (particle size 100 – 200 μ m) and 22 g $\pm$ 1 g of sulphuric acid (96 – 98 %) into a 200 ml Erlenmeyer flask. Shake until any lumps have disappeared. The mixture will heat up considerably.

Wear face protection and gloves.

Store the mixture in a closed desiccator over P₂O₅.

B.5.2.2 *Preparation of combined benzenesulfonic acid/sulphuric acid column*

Immediately prior to the sample preparation procedure put 0,5 g $\pm$ 0,05 g of the silica gel/sulphuric acid mixture onto the top of the 3 ml benzenesulphonic acid separating column. The silica gel/sulphuric acid mixture should be used within one week.

Follow the procedure in 11.1.

Annexe/Annex C
(informative)

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This Indian Standard has been developed from Doc No.: ETD 03 (6357).

Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

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